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(54) **Gas oil composition**

Dieselölzusammensetzung

Composition de gas-oil

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Description

The present invention relates to the use of a lubricity improving agent in a gas oil composition for motor vehicles (diesel fuel), with a low sulfur content.

Sulfur contained in gas oils (diesel fuels) constitutes a particularly serious environmental problem. New regulations have been discussed for long time at EC level, following other regulations, already adopted in such geographical regions as California and Sweden, which considerably limit the sulfur and aromatics contents in gas oil, which are thought to contribute to the emissions of polluting substances (SO_x , NO_x , particulates and smoke) in diesel engine exhaust gases.

Since 1985 Laws have been passed in California which limit to 0.05% by weight the allowed sulfur level in gas oil. Subsequently, in November 1990, EPA (Environmental Protection Agency), in accordance with EMA (Engine Manufacturers Associations), API (American Petroleum Institute) and NCFC (National Coalition of Farm Cooperatives), passed Laws applicable throughout the whole territory of the United States, which set limits both to sulfur content and to aromatics content in gas oil (maximal allowed level 35% by volume). Such regulations went into effect in October 1991.

Owing to a more deteriorated environmental situation, in California stricter regulations were passed by CARB (California Air Resources Board), which limit the aromatics content in gas oil to 10% by volume (for large size refineries with a production capacity of 50.000 DBP) and to 20% (for small size refineries). These regulations went into effect on October 1st, 1993. These regulations should allow the newly manufactured diesel engines to limit the particulates emissions to 0.10 g/bhph, versus the presently allowed threshold value of 0.25 g/bhph.

As regards the European Countries, Sweden passed regulations which, through strong tax relief policies, stimulate the production of ecological gas oils. For example, for metropolitan Stockholm area, gas oils have been subdivided into the following classes:

Gas oil type	Total Aromatics Content	Polynuclear Aromatics Content	Sulfur	Tax Relief
Class 1	< 5% v	< 0.1% v	< 10 ppm	35%
Class 2	< 20% v	< 1% v	< 50 ppm	15%
Class 3	< 25% v	-----	<500 ppm	0%

As regards the European Economic Community, only a short time ago regulations were passed and turned into effect, which limit the sulfur content in gas oils at no more than 0.2% by weight, and stricter regulations are being discussed at present, which should go into effect inuring from 1996. Such regulations should provide for sulfur level to be limited at 0.05% by weight, besides limiting the aromatics contents.

Waiting for stricter regulations, Italy, by means of a Ministry Decree, rendered mandatory, inuring from 1992, using, in metropolitan areas, gas oils containing 0.1% by weight of sulfur.

The decrease in sulfur and aromatics levels in gas oils is technically obtained by means of refining treatments, in particular by catalytic hydrogenation. However, it was observed that decreasing sulfur and aromatics levels in gas oils causes problems of damage of injection system components in diesel engines, which are due to the decreased lubricity of the fuel. In particular, it was observed that gas oils with a sulfur content equal to, or higher than, 0.2% by weight and an aromatics level of the order of 30% by weight do not cause any particular lubricity problems. However, when sulfur level decreases down to lower values than 0.2% by weight, and the aromatics level decreases down to lower values than 30% by weight, phenomena of wear of the injection pumps, in particular of rotary pumps and of pump injectors, arise with a proportionally increasing intensity. So, e.g., using Swedish gas oils of the above reported classes 1 and 2 causes the failure of a rotary pump of light-duty engines (i.e., car engines) after an average distance covered of about 10.000 km. In low-sulfur, low-aromatics gas oils, the gas oil capability is in fact lost or, at least, decreased, of supplying a proper lubrication, i.e., the capability of forming a film capable of keeping the surfaces of the mechanical components separated from each other during their movement relative to each other. Such a capability, referred to as "lubricity", also depends on the geometry and composition of the lubricated components and on the operating conditions.

In the art, the use is known of gas oil additives, usually understood as anti-wear agents, of the types of fatty acid esters, unsaturated dimerized fatty acids, primary aliphatic amines, fatty acid amides of diethanolamide and long-chain aliphatic monocarboxy acids, such as disclosed, e.g., in U.S. Patent Nos. 2,252,889; 4,185,594; 4,208,190; 4,204,481 and 4,428,182. Most of them are additives which display their desired characteristics within a range of relatively high concentrations, a feature which is particularly undesired, also on considering their costs. In U.S. Patent No. 4,609,376, anti-wear additives are disclosed, which are formed by esters of monocarboxy or polycarboxy acids and polyhydroxy alcohols. These additives are useful in alcohol containing fuels.

The present Applicant has now found, according to the present invention, that a particular class of alkyl esters of higher fatty acids of natural origin, generally formed by straight-chain, mono- or poly-unsaturated acids, are lubricity

improver additives which are highly effective in gas oils with low sulfur and aromatics contents. In particular, these types of esters are available as that product which is known on the market with the name "bio-diesel", which is basically constituted by a blend of methyl esters of fatty acids of vegetable origin. Bio-diesel, which was proposed for use as a low polluting diesel fuel, is a commercially available product and constitutes a very cheap additive, as compared to the

In accordance therewith, the present invention relates to the use of mixtures of C_1 - C_5 alkyl esters of saturated and unsaturated, straight chain fatty acids of from C_{12} to C_{22} carbon atoms, derived from vegetable oleaginous seeds, in an amount of from 100 to 10.000 ppm (parts per million parts by weight), in gas oil composition (diesel fuel) with a sulphur content equal to, or lower than about 0.2 percent by weight and with a content of aromatic hydrocarbons lower than about 30% by weight as a lubricity improving agent.

According to the present invention, the expression "lower alkyl esters" means C_1 - C_5 esters, in particular methyl and ethyl esters, with the methyl ester being preferred.

As already briefly mentioned hereinabove, the methyl esters of the saturated, mono- and poly-unsaturated, C_{16} - C_{22} , fatty acids, mixed with each other, are known on the market as "bio-diesel" or "rapeseed methyl ester" (RME), according to their origin, and were proposed in the past for use as low polluting diesel fuels.

Bio-diesel is normally obtained by starting from oleaginous seeds, in particular from rapeseed, sunflower and soy bean seeds. Said seeds are submitted to grinding and/or solvent extraction treatments (e.g., with n-hexane) in order to extract the oil, which is essentially constituted by triglycerides of saturated and unsaturated (mono- and poly-unsaturated, in mixture with each other, in proportions depending on the selected oleaginous seed), C_{16} - C_{22} , fatty acids. Said oil is submitted to a filtration and refining process, in order to remove any possible free fats and phospholipids present, and is finally submitted to a trans-esterification reaction with methanol in order to prepare the methyl esters of the fatty acids, which constitute bio-diesel.

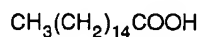
Typical physical characteristics of a bio-diesel are the following:

-- density (15°C)	0.84/0.90 g/ml
-- initial distillation point	min. 300°C
-- end distillation point	max. 400°C
-- flash point	min. 100°C
-- sulfur content	<0.01% by weight
-- viscosity (38.7°C)	3.5/5 cSt

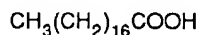
A typical elemental analysis of a bio-diesel yields the following results: carbon 77%; hydrogen 12%; and oxygen 11% by weight.

A typical composition of a bio-diesel derived from rape seed oil contains the methyl esters of the following C_{16} - C_{18} fatty acids at the following per cent by weight levels:

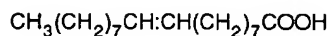
5% palmitic acid (hexadecanoic or cetyl acid)



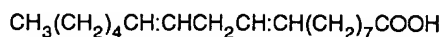
2% stearic acid (octadecanoic acid)



63% oleic acid (cis-octadecenoic acid)

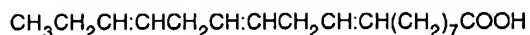


20% linoleic acid



9% linolenic acid (9,12,15-octadecatrienoic acid)

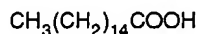
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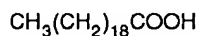
1% octadecatetraenoic acid

A typical composition of bio-diesel derived from sunflower oil, contains the methyl esters of the following C₁₆-C₂₂ fatty acids, as weight per cent values:

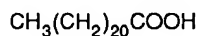
8 % palmitic acid (hexadecanoic or cetyl acid)



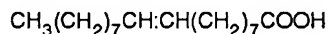
0.5% arachic acid (eicosanoic acid)



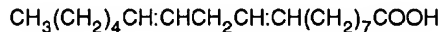
0.2% behenic acid (docosanoic acid)



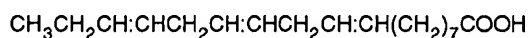
20 % oleic acid (cis-octadecenoic acid)



67.7% linoleic acid



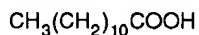
0.5% linolenic acid (9,12,15-octadecatrienoic acid)



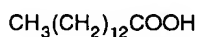
1 % octadecatetraenoic acid.

A typical composition of bio-diesel derived from soy bean oil contains the methyl esters of the following C₁₆-C₁₉ fatty acids, as weight per cent values:

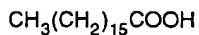
0.5% lauric acid



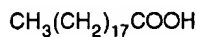
0.5% miristic acid



12 % heptadecanoic acid

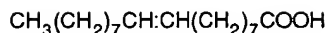


4 % nonadecanoic acid



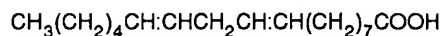
5

25 % oleic acid (cis-octadecenoic acid)



10

52 % linoleic acid



15

6 % linolenic acid (9,12,15-octadecatrienoic acid)



20

Of course, the higher alkyl esters of the above listed aliphatic carboxy acids, containing up to 5 carbon atoms in their alkyl moiety, can be used, although the methyl esters constitute the lubricity improver agents for low-sulfur, low-aromatics gas oils.

Therefore, the lubricity improver agent for diesel fuel, according to the present invention, is constituted by a mixture of lower alkyl esters, and preferably methyl esters, of a mixture of fatty acids with a C₁₂-C₂₂ straight chain, mainly with an even number of carbon atoms in their molecule, which mixture contains from 5 to 20% by weight of saturated fatty acids, from 70 to 95% by weight of total mono-unsaturated and di-unsaturated fatty acids, and from 0 to 10% by weight of total tri-unsaturated and tetra-unsaturated fatty acids.

The most important saturated fatty acids, present in bio-diesel as their methyl esters, are: lauric acid, palmitic acid and stearic acid. The most important unsaturated fatty acids, present in bio-diesel as their methyl esters, are: oleic acid, linoleic acid and linolenic acid.

Therefore, the lubricity improver agent, according to the present invention, will have a composition as indicated hereinabove, in which the saturated acids are constituted by one or more from among lauric acid, palmitic acid and stearic acid; the mono-unsaturated acids are essentially constituted by oleic acid, the di-unsaturated acids by linoleic acid and the tri-unsaturated acids by linolenic acid.

The lubricity improver agent will be applied to gas oils with a sulfur content lower than 0.2% by weight and preferably with a sulfur content lower than 0.1% by weight, up to reach sulfur-free, or essentially sulfur-free, gas oils, such as, e. g., gas oils containing 10 ppm, or less, of sulfur (corresponding to class 1 of Swedish gas oils, as reported hereinabove).

The concentration of the lubricity improver agent used in the compositions according to the present invention, will depend on sulfur concentration in gas oil, and, the lower the sulfur content, the higher, however within the above reported range, such a concentration will be. The present Applicant found anyway that, usually, an amount of improver agent of the order of 200-1,000 ppm is normally large enough in order to restore the desired lubricity, or even improve it, in gas oils containing 0.1-0.05% by weight thereof.

The gas oils which can be used according to the present invention, are gas oils for motor vehicles of petroleum origin, or gas oils produced by synthesis, or they are gas oils containing up to about 10% by volume of oxygen containing compounds, in particular of ether character, having, in any cases, a sulfur content equal to, or lower than, 0.2% by weight, and an aromatics content lower than 30% by weight.

Preferably, gas oils of petroleum origin are used, possibly admixed with usual additives, such as cetane number improvers, and agents which improve the low temperature properties of gas oil (e.g., pour point improvers, cloud point improvers and freezing point improvers). Typical specifications for gas oils are reported in the following table.

55

	A	B	C	D	E
GAS OIL					
Density 15°C, g/ml	0.81/0.86	0.82/0.86	0.82/0.86	0.80/0.82	0.80/0.82
Distillate at 150°C, % by vol.	max 2	max 2	---	---	---
Distillate at 250°C, % by vol.	25/<65	25/<65	---	---	---
Distillate at 350°C, % by vol.	min 85	min 85	min 90	100	100
Flash point, °C	min 55	min 85	---	---	---
Sulfur, % by weight	max 0.2	max 0.1	max 0.05	max 0.005	max 0.001
Cetane number	min 50	min 50	---	min 47	---
Viscosity at 37.8°C, cSt	2/5.35	2/5.35	---	---	---
Total aromatics, % by vol.	---	---	---	max 20	max 5
Polynuclear aromatics, % by vol.	---	---	---	max 1	max 0.1

Gas oil "A" is a typical EEC 1993 gas oil. Owing to its sulfur contents, normally the above mentioned lubricity problems do not exist. Gas oil "B" is a typical non-polluting EEC 1993 gas oil. Gas oil "C" is an EEC gas oil contemplated by the regulations due to be passed inuring from 1996, having a composition falling within the Swedish class 3 of gas

oils, as reported hereinabove. Gas oils "D" and "E" are gas oils falling within the scope of Swedish classes 2 and 1 for gas oils, as reported hereinabove. The gas oils of classes from "B" to "E", display lubricity problems and therefore are suitable for use in the compositions according to the present invention.

The compositions according to the present invention can be prepared by simply adding the lubricity improver agent to the selected gas oil. For the sake of use convenience, preparing and adding to gas oil concentrated solutions, e.g. containing 50% by weight of said improver agent in a liquid hydrocarbon solvent, which may advantageously be constituted by the same gas oil, may be convenient.

The lubricity of gas oils is determined according to the method proposed by LUCAS CAV Ltd., and derives from the standard ASTM method D 2783 used for evaluating the lubricity of lubricant oils. More particularly, the method is carried out by using the Four-ball E.P. Tribological Tester, which is capable of measuring lubricity in terms of load carrying capacity (L.C.C.), which expresses the maximal pressure under which the lubricating film, formed by the fuel, is capable of retaining such lubricity properties as to prevent deep roughening and surface seizure (scuffing) from taking place. The tester consists of four balls of 1,27 cm (1/2-inch) of diameter, wherein three of them, pressed against each other, remain in a stationary state inside the "ball-pot", with the centre of each of said balls being on a same horizontal plane and said balls being equidistant from the revolutionary tester axis. The fourth ball is above said three balls, and is mounted on a rotating chuck and is into lubricated contact with the underlying three balls, which cannot rotate. The machine load is supplied through a lever and weight system to the ball pot, i.e., to the three stationary balls, which are urged against the fourth, upper ball (therefore, the load is applied from bottom upwards). The contact (sliding) surface between the bottom balls and the fourth, upper ball, is always the same; on the three lower balls, a wear scar is formed, the diameter of which depends on the following variables: applied load (kg), fourth ball revolution speed (revolutions per minute), contact test time (seconds) and, of course, on the characteristics of the lubricant used. The size of the wear scar is measured under the microscope.

In the present testing, the following parameters were used:

- contact time per each single load = 10 seconds;
- revolution speed of the fourth ball = 1420 revolutions per minute;
- measurement of wear scar diameter = under microscope (accuracy ± 0.001 mm).

Sequential tests with higher and higher load values were carried out with new balls and the machine load was increased by a factor of 1.26 relatively to the lower load used in the preceding tests. The load was increased until a sudden decrease in end contact pressure (L.C.C.) was obtained, which is calculated by means of the following relationship:

$$P = 0.52L/d^2$$

wherein:

P is the end contact pressure expressed as kg/mm²,
d is the diameter of the wear scar (mm), and
L is the machine load (kg).

The load carrying capacity (L.C.C.) of a fuel is the maximal value of contact pressure which was obtained from a test series with increasing loads.

The following gas oils were tested:

- (I) Gas oil "A" containing 0.2% by weight of sulfur (reference gas oil);
- (II) Gas oil "B" containing 0.1% by weight of sulfur (comparison gas oil);
- (III) Gas oil "C" containing 0.05% by weight of sulfur (comparison gas oil);
- (IV) Gas oil "C" containing 0.05% by weight of sulfur and admixed with 500 ppm of bio-diesel from sunflower, having the composition as reported in the disclosure;
- (V) Gas oil "C" containing 0.05% by weight of sulfur and admixed with 1,000 ppm of bio-diesel from sunflower, having the composition as reported in the disclosure;
- (VI) Gas oil "C" containing 0.05% by weight of sulfur and admixed with 10,000 ppm of bio-diesel from sunflower, having the composition as reported in the disclosure;
- (VII) Low-polluting gas oil containing less than 0.1% by weight of sulfur (comparison gas oil);
- (VIII) Low-polluting gas oil containing less than 0.1% by weight of sulfur (VII) admixed with 1,000 ppm of bio-diesel from sunflower, having the composition as reported in the disclosure.

The performance of gas oils from (I) to (VIII), in terms of lubricity, are expressed as machine load (kg) and load carrying capacity (kg/mm²) and are reported in the following table.

Gas Oil No.	Load Carrying Capacity (kg/mm ²)	Machine Load (kg)
I	173.3	30
II	144.44	25
III	89.65	8
IV	173.3	30
V	173.33	30
VI	202.22	35
VII	115.15	20
VIII	202.22	35

It should be observed that those gas oils which display L.C.C. (load carrying capacity) values of round 100 kg/cm² are very likely riskful in terms of failure of mechanical components in diesel engines.

Claims

1. Use of mixtures of C₁-C₅ alkyl esters of saturated and unsaturated, straight chain fatty acids of from C₁₂ to C₂₂ carbon atoms, derived from vegetable oleaginous seeds, in an amount of from 100 to 10.000 ppm (parts per million parts by weight), in gas oil composition (diesel fuel) with a sulphur content equal to, or lower than about 0.2 percent by weight and with a content of aromatic hydrocarbons lower than about 30% by weight as a lubricity improving agent.
2. Use according to Claim 1, characterized in that said alkyl esters of fatty acids are methyl esters.
3. Use according to Claim 1, characterized in that said fatty acid esters are those known as "bio-diesel", "rapeseed methyl ester" or (RME), derived from soy bean, rapeseed or sunflower seeds oil.
4. Use according to Claim 1, characterized in that said esters are a mixture of esters of fatty acids with a C₁₂-C₂₂ straight chain, mainly with an even number of carbon atoms in their molecule, which mixture contains from 5 to 20% by weight of saturated fatty acids, from 70 to 95% by weight of total mono-unsaturated and di-unsaturated fatty acids, and from 0 to 10% by weight of total tri-unsaturated and tetra-unsaturated fatty acids.
5. Use according to Claim 4, characterized in that said saturated fatty acids are lauric acid, palmitic acid and stearic acid and said mono-, di- and tri-unsaturated acids respectively are oleic acid, linoleic acid and linolenic acid.
6. Use according to Claim 1, characterized in that the sulfur content in gas oil is equal to 0.1% by weight, or is lower than this value, down to complete or substantial absence of said sulfur.
7. Use according to Claim 1, characterized in that said gas oil is a gas oil for motor vehicles of petroleum origin, or of synthetic origin, or it may be a gas oil containing a level of up to about 10% by volume of oxygen containing compounds, in particular of ether character.
8. Use according to Claim 7, characterized in that said gas oil additionally contains one or more additives selected from among cetane number improvers and low temperature characteristics improvers of said oil.

Patentansprüche

1. Verwendung eines Gemisches von C₁-C₅-Alkylestern gesättigter und ungesättigter, geradkettiger Fettsäuren mit 12 bis 22 Kohlenstoffatomen, die von ölhaltigen Gemüsepflanzensamen abgeleitet sind, in einer Menge von 100 bis 10.000 ppm (Teile pro Million Gewichtsteile) in einer Dieselölzusammensetzung (Dieselkraftstoff) mit einem Schwefelgehalt von etwa 0,2 Gew.-% oder darunter und mit einem Gehalt an aromatischen Kohlenwasserstoffen unter etwa 30 Gew.-%, als ein das Schmiervermögen verbesserndes Mittel.

2. Verwendung nach Anspruch 1, dadurch gekennzeichnet, daß die Alkylester der Fettsäuren Methylester sind.
3. Verwendung nach Anspruch 1, dadurch gekennzeichnet, daß die Fettsäureester als "Biodiesel" als "Rapssamen-methylester" oder (RME) bekannt sind, die von Sojabohnen, Rapssamen oder Sonnenblumensamenöl abgeleitet sind.
4. Verwendung nach Anspruch 1, dadurch gekennzeichnet, daß die Ester ein Gemisch von Estern geradkettiger Fettsäuren mit 12 bis 22, hauptsächlich mit einer geraden Anzahl an Kohlenstoffatomen sind, wobei das Gemisch 5 Gew.-% bis 20 Gew.-% gesättigte Fettsäuren, 70 Gew.-% bis 95 Gew.-% insgesamt einfach und zweifach ungesättigten Fettsäuren und 0 Gew.-% bis 10 Gew.-% insgesamt dreifach und vierfach ungesättigte Fettsäuren enthält.
5. Verwendung nach Anspruch 4, dadurch gekennzeichnet, daß die gesättigten Fettsäuren Laurinsäure, Palmitinsäure und Stearinsäure sind und die einfach bzw. zweifach ungesättigten Fettsäuren Ölsäure, Linolsäure bzw. Linolensäure sind.
6. Verwendung nach Anspruch 1, dadurch gekennzeichnet, daß der Schwefelgehalt im Kraftstofföl gleich 0,1 Gew.-% ist oder unter diesem Wert bis zur vollständigen oder wesentlichen Abwesenheit des Schwefels liegt.
7. Verwendung nach Anspruch 1, dadurch gekennzeichnet, daß das Dieselöl ein Dieselöl für Kraftfahrzeuge aus Erdöl oder synthetischen Ursprungs ist oder ein Dieselöl mit einem Gehalt von bis zu etwa 10 Vol.-% an Sauerstoff enthaltenden Verbindungen, insbesondere mit Ether-Charakter.
8. Verwendung nach Anspruch 7, dadurch gekennzeichnet, daß das Dieselöl zusätzlich ein oder mehrere Additive enthält ausgewählt unter Octanzahl-Verbesserern und Verbesserern des Tieftemperaturverhaltens des Öls.

Revendications

1. Utilisation de mélanges d'alkylesters C_1-C_{25} d'acides gras saturés et insaturés à chaîne linéaire avec des atomes de carbone en C_{12} jusqu'à C_{22} dérivés de graines oléagineuses végétales dans une quantité de 100 à 10 000 ppm (parties par million en poids par parties), dans une composition de gasoil (diesel) avec une teneur en soufre égale à ou inférieure à environ 0,2 % en poids et avec une teneur en hydrocarbure aromatique inférieure à environ 30 % en poids en tant qu'agent améliorant l'aptitude à la lubrification.
2. Utilisation selon la revendication 1, caractérisée en ce que les alkylesters des acides gras sont des méthylesters.
3. Utilisation selon la revendication 3, caractérisée en ce que les esters des acides gras sont ceux connus comme "bio-diesel" "méthylester de colza" ou RME, dérivés de l'huile des graines de tournesol ou de colza ou de soja.
4. Utilisation selon la revendication 1, caractérisée en ce que les esters sont un mélange d'esters des acides gras avec une chaîne linéaire $C_{12}-C_{22}$ principalement avec un nombre pair d'atomes de carbone dans leur molécule, lequel mélange contient de 5 à 20 % en poids d'acides gras saturés, de 70 à 95 % en poids d'acides gras di-insaturés et mono-insaturés au total et de 0 à 10 % en poids d'acides gras tétra-insaturés et tri-insaturés au total.
5. Utilisation selon la revendication 4, caractérisée en ce que les acides gras saturés sont l'acide laurique, l'acide palmitique et l'acide stéarique et les acides mono-, di- et tri-insaturés respectivement sont l'acide oléique, l'acide linoléique et l'acide linolénique.
6. Utilisation selon la revendication 1, caractérisée en ce que la teneur en soufre dans le gasoil est égale à 0,1 % en poids ou inférieure à cette valeur jusqu'à l'absence totale ou sensible de soufre.
7. Utilisation selon la revendication 1, caractérisée en ce que le gasoil est un gasoil destiné aux véhicules à moteur d'origine essence ou d'origine synthétique ou peut être un gasoil contenant un niveau jusqu'à environ 10 % en volume d'oxygène contenant des composés en particulier du caractère éther.
8. Utilisation selon la revendication 7, caractérisée en ce que le gasoil contient de plus un ou plusieurs additifs sélectionnés à partir d'agents améliorant l'indice de cétane et des agents améliorant les caractéristiques faibles tem-

pératures de cette huile.

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